

X-RAY DIFFRACTION - XRD

Introduction

X-ray Diffraction (XRD) analyses allow the identification of minerals based on their atomic structure. During XRD analysis, x-ray beams are reflected off parallel atomic layers within a mineral over a range of diffraction angles. Because the x-ray beam has a specific wavelength, there are only specific angles which the exiting rays will be detected and counted by the detectors. Every substance has a unique diffraction pattern which can be used for identification. With this instrument, scientists can evaluate the mineralogical composition of sediments and the alteration products of ocean crust material.

XRD analyses were done on powdered samples. Quantitative analysis of a powdered sample of unknown composition is a difficult problem. An analysis program on the data acquisition computer could be run to analyze the results. After 1991, a freeware program for the Apple Macintosh platform called MacDiff was available to the scientists for a quick qualitative and quantitative analysis of samples. For more thorough analyses, scientists could take XRD files to their home institution.

Data Acquisition

Standard Operating Procedures

Samples were normally ground to a fine powder using a Spex mill or mortar and pestle when the sample was very small. The powder was pressed into a sample holder or smeared on a glass plate that was placed into the sample holder. Prior to Leg 147, XRD analyses were collected on a DEC PDP-11, with the files archived on floppy disks. Data analysis software could be used to create a printout of the diffraction peaks; occasionally, a printout of a spectral plot, or the quantitative analyses was also printed. Starting with Leg 147, the data acquisition software was moved to a PC-based computer with the raw XRD files written to disk.

Data Analysis

The analysis and interpretation of the XRD diffraction data were the responsibility of the Shipboard Scientific Party. Sometimes, the scientists on a cruise were not familiar with the interpretation of XRD data. For several ODP legs, there is no mention of XRD, even though samples were analyzed.

Archive

Pre-Janus Archive

Prior to Leg 147, XRD raw data files were copied onto DEC PDP-11 floppy disks. One copy remained on the ship, and one copy was archived at ODP/TAMU. The printouts that were made of the diffraction peaks and any plots were brought back to shore and microfilmed for archival storage. After the transition to the PC-based data acquisition, files were written to disk and transferred to shore and archived on ODP/TAMU servers.

The DEC PCP-11 system was deactivated, sent back to shore and eventually excessed. Unfortunately, the data stored on those DEC floppies could not be retrieved due in part because the computer system was not capable of being put onto a network. The raw data for the XRD analyses before Leg 147 were no longer accessible.

Migration of XRD data to Janus

The data model for X-Ray Diffraction data can be found in Appendix I. Included are the relational diagram and the list of the tables that contain data pertinent to XRD analyses, the column names and the definition of each column attribute. ODP Information Services Database Group was responsible for the migration of pre-Leg 171 data to Janus.

Archival of XRD data in Janus consisted of extracting the header information into a table - the header information from the XRD data file documented the system setup and data acquisition parameters. Then, the entire raw data file was copied line by line into a table designed to hold the text of the data files. Migration of data for Legs 147 – 170 was done in the same manner.

Pre-Leg 147 data could not be uploaded due to the lack of data files. Advances in scanning technology gave ODP/TAMU Information Services the opportunity to scan the pre-Leg 147 printouts and upload those images to the database as Primedata Images.

Janus X-Ray Diffraction Format

X-ray diffraction raw data files can be retrieved from Janus Web using a predefined query. The X-Ray Diffraction (XRD) query webpage allows the user to extract data using the following variables to restrict the list of available data files: leg, site, hole, core, section, depth, or latitude and longitude ranges. Table 1 lists the data that are retrieved from the XRD query. To extract the data file, the user must click on the *Data File* link. Currently, there is no other way to download all files of interest from Janus Web. The user can contact the IODP Data Librarian for help when requesting a large number of the XRD data files [database@iodp.tamu.edu].

Appendix II contains information about the format of the archived ASCII files. There are three examples of XRD data files. The header format used from Leg 147 through Leg 164 is listed under Appendix II-B. The header format used from Leg 165 through

Leg 180 is listed under Appendix II-C. And the header format Leg 181 through Leg 210 is listed under Appendix II-D.

The name of the file in the archived ASCII set is the full sample identification. The first line of each data file contains an abbreviated sample identification text string. For samples from Legs 147-180, that identification was manually entered by the operator or scientist. Usually, there is no *Leg* identification and *Site* number may be shortened. Starting with Leg 181 the database-assigned Sample_ID was inserted in the first line in order to ensure the data were being linked with the correct sample and speed the upload of files to Janus,. This change made it very difficult to determine any information about the sample by looking at the first line of the file.

Table 1. X-Ray Diffraction (XRD) query

Item Name	Janus Table	Janus Column Name
Leg	SECTION	Leg
Site	SECTION	Site
Hole	SECTION	Hole
Core	SECTION	Core
Coretype	SECTION	Core_type
Section	SECTION	Section_number
Top (cm)	SAMPLE	Top_interval x 100
Bottom (cm)	SAMPLE	Bottom_interval x 100
Depth (mbsf)	DEPTH_MAP, SAMPLE	DEPTH_MAP.Map_interval_top + SAMPLE.Top_interval
Data File	XRD_FILE	Line_Text
Run	XRD_HDR_DATA	XRD_run_id
Comment	XRD_HDR_DATA	XRD_comment

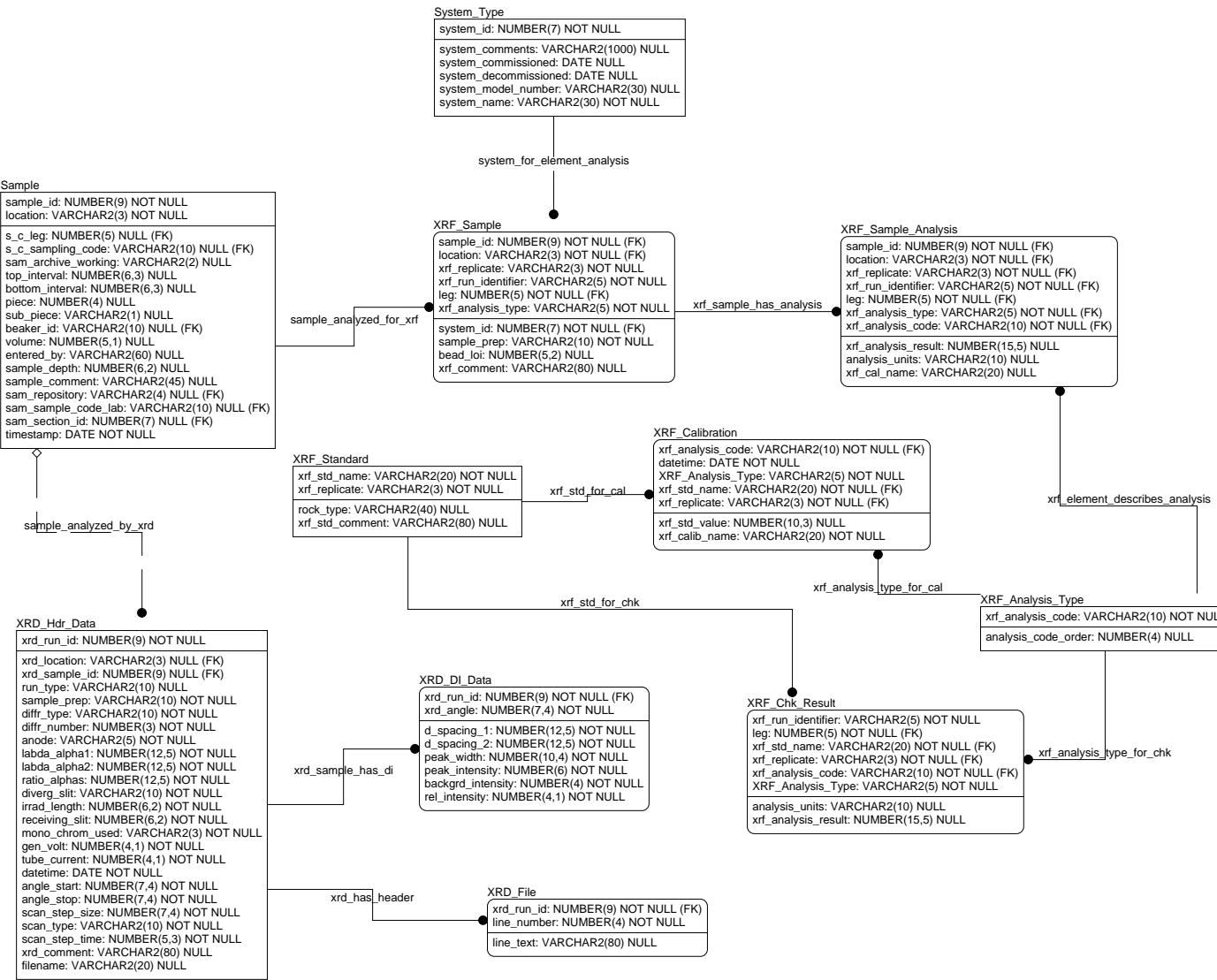
Data Quality

Almost 16,000 XRD analyses were done on samples during the ODP. It is unknown how many have been analyzed for their mineral content.

The modification of the sample identifier in the first line of the XRD file significantly improved the accuracy and integrity of the XRD data in Janus, but after the file has been retrieved from the database, the user must be careful to document the file in such a way that its location is not lost. [The **Sample Report** query on Janus Web does retrieve Sample_ID, but there is currently not a way to enter a Sample_ID and have the query return its location information.]

Reference

Rangin, C., Silver, E., von Breymann, M.T., et al., 1990. Explanatory Notes, in *Proc. ODP, Init. Repts.*, 124: College Station, TX (Ocean Drilling Program), p. 28.



APPENDIX I: Janus Data Model – X-Ray Diffraction – XRD

X-Ray Diffraction - XRD		
Table Name	Column Name	Column Comment
XRD_Hdr_Data	xrd_run_id	Oracle-generated sequence number that uniquely identifies an XRD analysis
	xrd_location	Code that indicates which Janus application assigned the sample_id. Values are SHI(ship), GCR (Gulf Coast Repository), ECR (East Coast Repository, WCR (West Coast Repository) and BRE (Bremen repository). Used with xrd_sample_id to uniquely identify a sample.
	xrd_sample_id	Oracle-generated sequence number that with xrd_location uniquely identifies a sample.
	run_type	Type of run - Sample, Control-1, Control-2 and Control-3 -- are current run types in system. New run types will be added.
	sample_prep	The type of preparation used for a sample. For XRD samples - bulk sample, clay separation, glycolated or heated.
	diffrr_type	The type of diffractometer used for the XRD analysis. ODP used Philips PW1710.
	diffrr_number	The XRD can control multiple diffractometers. However, on the ship the XRD was configured to run only one diffractometer in the first position.
	anode	The anode of the XRD tube, such as Copper (Cu).
	labda_alpha1	Wavelength output of the XRD tube, in angstroms.
	labda_alpha2	Wavelength output of the XRD tube, in angstroms
	ratio_alphas	Ratio of the alphas for an XRD tube.
	diverg_slit	The type of divergence slit used for an XRD analysis. If an automatic divergence slit is used, the irradiated length is listed in millimeters
	irrad_length	The irradiation length for the XRD in millimeters. (only applies if an automatic divergence slit is used).
	receiving_slit	Receiving slit for the XRD, in millimeters
	mono_chrom_used	Monochromator used, yes or no
	gen_volt	Voltage applied to the XRD tube, in kilovolts.
	tube_current	Current applied to the XRD tube in millamps.
	datetime	Date/time stamp for the XRD analysis.
	angle_start	The angle of the XRD goniometer at the start of an analysis, in degrees.
	angle_stop	The angle of the XRD goniometer at the end of an analysis, in degrees.
	scan_step_size	The step size of the XRD goniometer during an analysis, in degrees 2 theta.
	scan_type	The type of scan used during an XRD analysis, either continuous or step.
	scan_step_time	Time spent counting each step of an XRD run in seconds.
	xrd_comment	Comment related to a XRD sample
	filename	The filename of the XRD file generated from the Philips system. The filename is stored along with the header data for batch uploads because the barcode identifier for the sample may not be unique.
XRD_File		
XRD_File	xrd_run_id	Oracle-generated sequence number that uniquely identifies an XRD analysis
XRD_File	line_number	The line number of the XRD file.
XRD_File	line_text	The text contained in a line of header or data.
XRD_DI_Data		
XRD_DI_Data	xrd_run_id	Oracle-generated sequence number that uniquely identifies an XRD analysis
XRD_DI_Data	xrd_angle	Degrees 2-theta of a peak from a XRD diffractogram.
XRD_DI_Data	d_spacing_1	D-spacing associated with a mineral from an XRD analysis.
XRD_DI_Data	d_spacing_2	D-spacing associated with a mineral from an XRD analysis.
XRD_DI_Data	peak_width	Width of the peak from a XRD diffractogram, in degrees 2-theta.
XRD_DI_Data	peak_intensity	Intensity of a measured peak from a XRD diffractogram, in counts.
XRD_DI_Data	backgrd_intensity	Intensity of the background from a XRD sample.
XRD_DI_Data	rel_intensity	The intensity of a peak relative to the highest intensity peak for the scan in an XRD analysis.
Section		
Section	section_id	Unique Oracle-generated sequence number to identify each section. This is done because of the physical subsection / zero section problems. In adding new sections, deleting sections or changing sections - don't want to have to renumber.
Section	leg	Number identifying the cruise for which data were entered into the database.
Section	site	Number identifying the site from which the core was retrieved. A site is the position of a beacon around which holes are drilled.

	hole	Letter identifying the hole at a site from which a core was retrieved or data were collected.
	core	Sequential numbers identifying the cores retrieved from a particular hole. Cores are generally 9.5 meters in length, and are numbered serially from the top of the hole downward.
	core_type	A letter code identifying the drill bit/coring method used to retrieve the core. The coretype is only reported in the post-leg 113 processed data file.
	section_number	Cores are cut into 1.5 m sections. Sections are numbered serially, with Section 1 at the top of the core.
	section_type	Used to differentiate sections of core (S) from core catchers (C). Previously core catchers were stored as section CC, but in Janus core catchers are given the next sequential number from the last section recovered.
	curated_length	The length of the section core material, in meters. This may be different than the liner length for the same section. Hard rock cores will often have spacers added to prevent rock pieces from damaging each other.
	liner_length	The original length of core material in the section, in meters. Sum of liner lengths of all the sections of a core equals core recovery.
	core_catcher_stored_in	Sometimes the core catcher is stored in a D tube with a section. core_catcher_stored_in contains the section number of the D tube that holds the core catcher.
	section_comments	Comments about this section

Sample	sample_id	Oracle-generated sequence number that with location uniquely identifies a sample.
	location	Code that indicates which Janus application assigned the sample_id. Values are SHI (ship), GCR (Gulf Coast Repository), ECR (East Coast Repository), WCR (West Coast Repository) and BCR (Bremen Core Repository). Used with sample_id to uniquely identify a sample.
	s_c_leg	Number identifying the cruise for which data were entered into the database. Foreign key used with s_c_sampling_code to link samples with a scientist's sample request.
	s_c_sampling_code	Code used to identify samples taken for a sample request. Used with s_c_leg.
	sam_archive_working	Part of section where sample was taken. Valid values: WR – whole round, A – archive half, W – working half.
	top_interval	Distance in meters from the top of the section to the top of the sample.
	bottom_interval	Distance in meters from the top of the section to the bottom of the sample.
	piece	Additional identifier for hard rock samples. Each individual piece of rock within a section is numbered consecutively starting at the top of the section.
	sub_piece	Additional identifier for hard rock samples. When a piece is broken, the individual fragments are given consecutive letter designations. Note that subpiece assignments must be made in conjunction with piece numbers.
	beaker_id	The number on the moisture density beaker. Used for samples analyzed for moisture and density.
	volume	Volume of sample.
	entered_by	Indicates who entered the sample into the database.
	sample_depth	Depth of the sample.
	sample_comment	Comment about the sample.
	sam_repository	Repository where sample was taken. Valid values SHIP (ship), GCR (Gulf Coast Repository), ECR (East Coast Repository), WCR (West Coast Repository) and BCR (Bremen Core Repository).
	sam_sample_code_lab	Code to indicate the shipboard lab that will perform the initial analysis.
	sam_section_id	Unique Oracle-generated sequence number to identify each section. This is a foreign key that links a sample to leg, site, hole, core, and section.
	timestamp	Date and time when sample was entered into database. Samples taken before November 25, 1998 and migrated samples have the timestamp 11/25/1998 12:26PM.

System_Type	system_id	Unique identifier for a system of equipment used to collect data.
	system_comments	Comments associated with a piece of analytical equipment
	system_commissioned	Date that a piece of equipment was deployed to collect scientific data for the ODP.
	system_decommissioned	Date that a piece of analytical equipment was no longer used by the ODP.
	system_model_number	The model number of a piece of equipment used for scientific analysis.
	system_name	The name for a piece of equipment used for analysis.

Appendix II-A. Description of data items from X-Ray Diffraction Query.

Item Name	Column Description	Format
Leg	Number identifying the cruise. The ODP started numbering the scientific cruises of the <i>JR</i> at Leg 101. A leg was nominally two months duration. During the 18+ years of the ODP, there were 110 cruises on the <i>JR</i> .	Integer 3
Site	Number identifying the site. A site is the location where one or more holes were drilled while the ship was positioned over a single acoustic beacon. The <i>JR</i> visited 656 unique sites during the course of the ODP. Some sites were visited multiple times, including some sites originally visited during the Deep Sea Drilling Program for a total of 673 site visits.	Integer 4
Hole	Letter identifying the hole. Multiple holes could be drilled at a single site by pulling the drill pipe above the seafloor, moving the ship some distance away and drilling another hole. The first hole was designated 'A' and additional holes proceeded alphabetically at a given site. Location information for the cruise was determined by hole latitude and longitude. During ODP, there were 1818 holes drilled or deepened.	Text 1
Core	Cores are numbered serially from the top of the hole downward. Cored intervals are up to 9.7 m long, the maximum length of the core barrel. Recovered material was placed at the top of the cored interval, even when recovery was less than 100%. More than 220 km of core were recovered by the ODP.	Integer 3
Coretype	All cores are tagged by a letter code that identifies the coring method used.	Text 1
Section	Cores are cut into 1.5 m sections in order to make them easier to handle. Sections are numbered serially, with Section 1 at the top of the core. XRD analyses were made on samples taken from the sections. Core Catcher sections identified as "CC."	Integer 2 or Text 2
Top (cm)	The top interval of a measurement in centimeters measured from the top of a section.	Decimal F4.1
Bottom (cm)	The location of the bottom of a sample in centimeters measured from the top of a section.	Decimal F4.1
Depth (mbsf)	Distance in meters from the seafloor to the sample location.	Decimal F7.3
Filename	Name of raw data file that contains results of XRD analysis.	Text 60

Appendix II-B. XRD Header format used for data collected on Legs 147-164.

```

SampleIdent,894A 1R2 130-135 GLY,/
DiffType,PW1710,/
DiffNumber,1,/
Anode,Cu,/
DivergenceSlit,Automatic, 12,/
MonochromatorUsed,YES ,/
GeneratorVoltage, 40,/
TubeCurrent, 35,/
FileDateTime, 8-dec-1992 23:58,/
DataAngleRange, 2.0000, 23.9900,/
ScanStepSize, 0.010,/
ScanType,CONTINUOUS,/
ScanStepTime, 1.00,/
RawScan
272, 240, 225, 269, 234, 243, 196, 213
243, 231, 199, 202, 204, 188, 193, 222
166, 185, 199, 182, 159, 169, 182, 166
...
169, 159, 193, 166, 164, 182, 177, 185
/

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Appendix II-C. XRD Header format used for data collected on Legs 165-180.

```
SampleIdent,49A10X314,/  
Title1,Ocean Drilling Program,/  
Title2,PC-APD Software Version 3.6f,/  
DiffractionType,PW1710,/  
DiffractionNumber,1,/  
Anode,Cu,/  
LabdaAlpha1, 1.54056,/  
LabdaAlpha2, 1.54439,/  
RatioAlpha21, 0.50000,/  
DivergenceSlit,Automatic, 12,/  
ReceivingSlit,0.2,/  
MonochromatorUsed,YES ,/  
GeneratorVoltage, 40,/  
TubeCurrent, 35,/  
FileDateTime,22-Jan-1997 1:39,/  
DataAngleRange, 2.0000, 60.0000,/  
ScanStepSize, 0.020,/  
ScanType,STEP,/  
ScanStepTime, 1.00,/  
RawScan  
    17,     15,     9,     9,    18,    11,    13,    19  
    15,     13,     20,    11,    18,    15,    17,     8  
    19,     9,     17,    23,    18,    15,    11,    24  
    ...  
    32,     19,     35,    35,    26, /
```

Appendix II-D. XRD Header format used for data collected on Legs 181-210.

```
SampleIdent,ss001552434 bulk,/  
Title1,,/  
Title2,PC-APD Software Version 3.6f,/  
DiffractionType,PW1710,/  
DiffractionNumber,1,/  
Anode,Cu,/  
LabdaAlpha1,1.54056,/  
LabdaAlpha2,1.54439,/  
RatioAlpha21,.5,/  
DivergenceSlit,Automatic,12,/  
ReceivingSlit,.2,/  
MonochromatorUsed,YES,/br/>GeneratorVoltage,40,/  
TubeCurrent,35,/  
FileDateTime,10-Aug-2003 06:56,/  
DataAngleRange,2,70,/  
ScanStepSize,.02,/  
ScanType,STEP,/  
ScanStepTime,1,/  
Rawscan,  
    365,    342,    361,    369,    313,    361,    310,    310  
    339,    313,    303,    328,    328,    350,    299,    286  
    289,    256,    276,    289,    279,    313,    266,    282  
    ...  
    193, /
```